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S. W. STRATTON, DIRECTOR

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## DETERMINATION OF CELLULOSE IN RUBBER GOODS

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*Bureau of Standards*

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# DETERMINATION OF CELLULOSE IN RUBBER GOODS

By S. W. Epstein and R. L. Moore

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## I. INTRODUCTION

The chemist who still determines the rubber content of mixings by the difference method finds himself confronted from time to time with conditions that make an analysis by this method worthless. He finds ingredients present which are driven off in the ashing, yet are not included in the other determinations so that, unless these ingredients are determined separately, the figure for rubber hydrocarbon must necessarily be erroneous. One outgrowth of this condition was the development of a method for the determination of free carbon in rubber goods by A. H. Smith and S. W. Epstein of this Bureau.<sup>1</sup> Likewise it was found necessary to determine nitrogen by the Kjeldahl method in order to ascertain the quantity of glue in mixings. A great variety of rubber products which have cellulose impregnated with rubber in such a way that it can not be separated from it, come to the laboratory for analysis. Rubber sheeting, raincoat materials, balloon fabrics, and the wide variety of spread goods and frictioned fabrics represent a few of these. In addition there is the type of mixing which has fiber distributed throughout the compound, such as fiber soles, special light compounds, special packings. In order properly to evaluate these products it is evident that the fiber must be determined separately.

Further, the determination of cellulose in rubber presents a matter of primary importance to those engaged in checking up factory processes as well as those who are interested in competitors' products with the idea of duplication. The determination

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<sup>1</sup> Technologic Paper 136, U. S. Bureau of Standards.

of cellulose gives a direct method to ascertain the amount of rubber compound which has been spread or calendered upon the fabric. It gives a ready method of studying frictioned stocks. Finally, in the reclaiming industry it is important to know whether or not the reclaiming process in a particular case removes all or a part of the fiber present.

## II. DISCUSSION OF PROPOSED METHOD

B. D'Porritt in his "Isolation and Examination of Rubberized Fabrics"<sup>2</sup> obtains the weight of fabric by dissolution of the rubber coating in paraffin. In cases where there are fillers, these are removed by special treatment before the fabric is weighed. In the presence of fillers not soluble in dilute acids, he determines the weight of cellulose by combustion. It has been our experience that considerable organic matter is left insoluble when solution of rubber is attempted by means of paraffin. It seems to us, therefore, that the results of the combustion could not be very reliable for this reason alone, without considering gas black which may be present and the possible varying nature of the fabric. The only method in the literature that we could find which makes any claims toward analyzing a vulcanized rubber compound for its cellulose content is one by H. G. Hillen,<sup>3</sup> who was interested in finding out whether or not the reclaiming process left considerable quantities of cellulose in the finished product. He ground the rubber sample extremely fine by special means, and allowed it to remain in copper-ammonia solution for from 6 to 10 hours. It was then filtered and the cellulose precipitated from the filtrate by the addition of sulphuric acid. This was then filtered out and weighed. Hillen stated that (1) only high-grade compounds can be analyzed by this method; (2) the sample must be ground very fine by special means in order to be reasonably certain that all of the cellulose will be removed from the rubber; (3) unless care is taken to keep the ammonia from being given off during the period of 6 to 10 hours, there is considerable danger of error; (4) there is danger of the cellulose being hydrolyzed if it is left in contact with the sulphuric acid for any length of time; (5) it is difficult to filter the voluminous, gelatinous precipitate of cellulose since it clogs up the Gooch filter. In conclusion he stated that the procedure is accurate enough only for industrial purposes.

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<sup>2</sup> Rubber Age and Tire News, 9, p. 386; Aug. 10, 1919.

<sup>3</sup> Gummi-Zeitung, Mar. 3, 1916.



In our opinion the method of Hillen could not be acceptable because of the presence of rubber, substitutes, and other organic material during the immersion in copper-ammonia. It is not improbable that precipitates are obtained, when the sulphuric acid is added, that are other than cellulose. But what is more important, it is an uncertain thing to attempt to remove cellulose from a rubber compound with which it has been intimately mixed by a process which can be likened unto extraction. Therefore it was decided that the rubber must first be removed before any attempt could be made to determine the cellulose by any procedure. The most convenient way to accomplish the removal of rubber from the sample was found to be through the use of cresol. Early experimentation with cresol (B. P.  $198^{\circ}\text{C}$ ) had indicated that it would effect this solution at  $160$  to  $185^{\circ}\text{C}$  in from 2 to 4 hours, depending on the coefficient of vulcanization, and that the resulting solution after it had been diluted with petroleum ether (B. P. about  $45$  to  $50^{\circ}\text{C}$ ) would allow the fillers to settle out in a very short length of time, after which it could be filtered easily and rapidly.

Samples of cotton, free from moisture, were immersed in cresol and allowed to remain for the maximum period of 4 hours at  $185^{\circ}\text{C}$ . The solution was diluted with petroleum ether and filtered. The weight of the washed and dried cellulose after this treatment was identical with the weight of sample taken.

The use of cresol was therefore adopted to remove the rubber as the first step in the method as outlined. From this point we proposed to remove the fiber, and determine it as the loss in weight occasioned by the process of solution. Obviously, all mineral fillers which would be affected by the reagents used to dissolve the cellulose would have to be removed beforehand. Likewise the reagents used to remove these fillers must not affect the cellulose.

It was found that 10 per cent hydrochloric acid when used to wash cellulose would not affect it in such a way as to produce a change in weight. However, if acid of higher concentration was used, the effect was considerable, even to the extent of causing a loss of 10 per cent of its weight. The 10 per cent hydrochloric acid removed a large part of the fillers in most cases so that the residue consisted of the practically insoluble fillers and the fiber. The choice of a procedure to remove the cellulose and record the loss in weight as cellulose with accuracy had to be directed from a

knowledge of a possible effect on the insolubles and the Gooch asbestos. Concentrated hydrochloric acid, concentrated sulphuric acid and strong sodium hydroxide had to be ruled out because of this. It was decided to rule out the copper-ammonia solution because of the uncertainty of completely dissolving the cellulose, and because of the comparatively long time required. The final result of consideration of the various cellulose solvents led conclusively to the choice of acetic anhydride. It was found that acetic anhydride to which has been added a small quantity of concentrated sulphuric acid, dissolves cellulose with surprising rapidity. This solution will be complete in 5 to 10 minutes even when large quantities of substance are treated. Our early experiments indicated that after 10 per cent hydrochloric acid had been applied to the fillers obtained from a rubber mixing, the acetylation mixture would have no effect upon the residue. This was confirmed in later work.

Samples of dried cotton were treated with varying mixtures of acetic anhydride and sulphuric acid at different temperatures, and the time was noted in which complete solution took place. The resulting solutions of cellulose were filtered through weighed Gooch crucibles, and the pads washed with hot 90 per cent acetic acid and then with acetone. In every case where there seemed to be complete solution, the weight of the Gooch before filtration and after was identical. A direct result of this work was the evidence that 90 per cent acetic acid and acetone removed all of the acetylation products from the pad. It was found that a mixture of acetic anhydride and concentrated sulphuric acid made up 30:1 was the best adapted for the acetylation and that as much as 1 g of cotton fiber could be dissolved in 20 cc of this mixture in less than 15 minutes at a temperature of 75° C.

Some difficulty was encountered with the first attempts to acetylate material directly in the Gooch crucible. The cause was traced directly to the loss of small quantities of finely divided fillers in the manipulation. As a result, several refinements had to be introduced. The asbestos pad was removed from the Gooch crucible, placed in a weighing bottle, dried, and weighed. The material was then transferred to the beaker in which the acetylation was to take place. After the reaction was completed, the solution was filtered through a freshly prepared Gooch containing a thick pad of asbestos. The liquid filters very easily, so that the thick pad does not slow up the operation. By carrying out the work as given it was found that no particles of material would run through.



### III. DETAILS OF METHOD AS PROPOSED

After having considered the essentials of a procedure for the determination of cellulose in rubber goods, the following method was devised:

Digest a 0.5 g sample of rubber in a 250 cc assay flask with 25 cc of freshly distilled cresol (B. P.  $198^{\circ}\text{C}$ ) for 4 hours at 160 to  $185^{\circ}\text{C}$ . The digestion can be carried out on an electric hot plate, or better still in a constant-temperature oven. Allow the cresol to cool completely, and add 200 cc of petroleum ether (B. P. 45 to  $50^{\circ}\text{C}$ ) very slowly and with constant agitation. After the solution has settled completely and the supernatant liquid is perfectly clear, filter through a Gooch crucible containing a fairly thick pad of acid-treated and ignited asbestos, and wash 3 times with petroleum ether. Wash at least 5 times with hot benzene and then once or twice with acetone. Treat the contents of the flask with hot 10 per cent solution of hydrochloric acid, and transfer the entire contents of the flask to the Gooch crucible with the aid of a "policeman." Care must be taken in adding the first portion of acid to the Gooch, since, in the presence of carbonates, the effervescence which ensues may cause some of the material to be lost. It is best to add the acid a few drops at a time until there is no more effervescence. Continue to treat with hot 10 per cent solution of hydrochloric acid until the pad has been washed at least 10 times. Wash the pad free from chlorides with boiling water, and run small portions of acetone through it until the filtrate comes through colorless. Treat with a mixture of equal parts of acetone and carbon bisulphide until the solvent is no longer colored. Wash with alcohol, and dry for 1 hour and 30 minutes at  $105^{\circ}\text{C}$ . Remove the pad from the crucible with the help of a pair of sharp-pointed tweezers, and place it in a weighing bottle that is large enough to contain a 25 cc Gooch crucible or the size crucible that is being used. Use the underneath portions of the pad as a swab to clean the sides of the crucible. If necessary, moisten this asbestos with a little alcohol, since this will facilitate the complete removal of material which sometimes adheres tenaciously. Place the weighing bottle and contents in the drying oven for about 15 minutes, cool and weigh. Call this weight A.

Transfer the contents of the weighing bottle to a 50 cc beaker, taking precautions that no material is lost during the transfer, or allowed to remain in the weighing bottle. Add 15 cc of acetic anhydride and 0.5 cc of concentrated sulphuric acid, and allow to

digest for 30 minutes at 75° C. The steam bath can be conveniently used for this purpose. After the mixture has cooled completely dilute with 25 cc of 90 per cent acetic acid and filter through a weighed Gooch containing a thick pad of properly prepared asbestos. To guard against traces of material being carried through it is absolutely essential that this filtration, as well as the ones to follow, be very slow, and that only gentle suction be used. Wash with hot 90 per cent acetic acid until the filtrate comes through absolutely colorless, and then wash at least 4 times more. Wash about 5 times with acetone. After having taken care that all of the material has been washed out of the beaker in which the acetylation took place, remove the crucible from the funnel, clean the outside thoroughly, place it inside of the same weighing bottle that was used to weigh the pad, and dry for 2 hours at 150° C. Cool and weigh. Call this weight *B*.

Weight of Gooch crucible + weight *A* - weight *B* = loss due to acetylation or cellulose.

NOTE.—It is essential that all reagents given above be of chemically pure quality, and be filtered before using.

When large numbers of determinations were carried out according to the above procedure, it was found practical to distil the filtrates, and in this way recover a large part of the solvents used. The combined filtrates containing petroleum ether, benzene, and cresol were distilled on the steam bath to recover the petroleum ether. The benzene was recovered as the temperature was raised. Finally, the water condenser was replaced by an air condenser and the cresol distilled out at about 195 to 200° C. This distillate was usually only slightly yellow.

Likewise the filtrates from the acetylation, which contain acetic anhydride, acetic acid, and small quantities of sulphuric acid, were collected and distilled. The distillate was collected between 105 and 115° C, and was used for washing to take the place of the 90 per cent acetic acid called for in the proposed method.

#### IV. RESULTS OF ANALYSIS

In order to ascertain the reliability of the method as proposed a number of representative compounds were prepared, and the cellulose content determined by means of it. In the preparation of these compounds the fiber as well as all of the fillers were dried before weighing out the batch.

## Stocks Containing No Cellulose

G-2:	Per cent	B S. 53—Continued.	Per cent
Para.....	43.0	Sulphur.....	2.0
Glue.....	7.0	Lampblack.....	4.0
Whiting.....	35.0	Fiber.....	None.
Lampblack.....	7.0		<u>100.0</u>
Sulphur.....	3.0	S. B. 5:	
Litharge.....	3.0	Plantation crêpe.....	60.0
Magnesia.....	2.0	Golden antimony.....	5.0
Fiber.....	None.	Zinc oxide.....	10.0
	<u>100.0</u>	Lithopone.....	10.0
		Thiocarbanilide.....	2.0
B. S. 53:		Sulphur.....	3.0
First latex.....	48.0	Iron oxide.....	10.0
Zinc oxide.....	22.0	Fiber.....	None.
Litharge.....	24.0		<u>100.0</u>

Blank determinations made on the above mixings showed losses due to acetylation of 0.0006 g, 0.0007 g, and 0.0006 g, respectively.

The results obtained indicate that of the material which was insoluble in 10 per cent hydrochloric acid, and was present throughout the acetylation, nothing was dissolved by the acetylation reagents. Evaporation and ignition of the filtrate revealed no residue. It is evident that the method will not give misleading results, and that no cellulose will be indicated by it when it is not present.

## Stocks Containing Cellulose in the Form of Cotton Fiber

C-1:	Per cent	C-3:	Per cent
Smoked sheet.....	50.0	Plantation.....	59.0
Zinc oxide.....	30.0	Gas black.....	5.0
Whiting.....	6.0	Zinc oxide.....	30.0
Chrome green.....	3.0	Sulphur.....	5.0
Sulphur.....	3.0	Cotton fiber.....	1.0
Cotton fiber.....	8.0		<u>100.0</u>
	<u>100.0</u>	C-4:	
C-2:		First latex.....	40.0
Smoked sheet.....	70.0	Mineral rubber.....	5.0
Golden antimony.....	15.0	Gas black.....	15.0
Calcium sulphate.....	4.0	Zinc oxide.....	30.0
Sulphur.....	6.0	Magnesium oxide.....	2.0
Cotton fiber.....	5.0	Magnesia, light.....	3.0
	<u>100.0</u>	Sulphur.....	2.0
		Cotton fiber.....	3.0
			<u>100.0</u>

## By Analysis

	Per cent		Per cent
C-1: Cellulose.....	7.96	C-3: Cellulose.....	0.98
C-2: Cellulose.....	4.86	C-4: Cellulose.....	2.93



The above results indicate clearly without further discussion that by this method it is easy to determine widely varying amounts of cotton fiber very accurately even when accompanied by a miscellaneous collection of mineral fillers in the rubber compound. Attention is called to C-4, where 15.0 per cent of gas black was present. It was possible to get good figures on cellulose because the weighings were all made in weighing bottles and, therefore, no error was introduced by the absorption of quantities of moisture by the large amount of carbon. It was found to be absolutely impossible to get reliable results at all unless this was strictly observed.

In order to illustrate how the method applies to the checking up of calendered goods, two samples of fabric were run through the calender in such a way that the final products should be as nearly 30 and 50 per cent cloth, respectively, as possible.

The sample which was intended to be 30 per cent fabric analyzed 29.1 per cent cellulose.

The sample which was intended to be 50 per cent fabric analyzed 48.5 per cent cellulose.

Seven reclaims of unknown cellulose content were analyzed, and a great variation in the cellulose content of the different grades was uncovered. The results of analysis ranged from 0.8 per cent to the high value of 11.36 per cent. The wide range of values furnishes conclusive evidence of the importance of checking up the cellulose content of reclaims by both the reclaimer and the purchaser.

**Results of Determinations of Cellulose in Reclaims**

Reclaim	Cellulose content		
	Per cent	Per cent	Per cent
No. 1.....	11.36	11.36	11.34
No. 2.....	8.20	8.22	8.32
No. 3.....	3.18	3.22	.....
No. 4.....	3.30	3.26	.....
No. 5.....	3.24	3.24	.....
No. 6.....	5.2	5.0	.....
No. 7.....	0.8	0.74	.....

The presence of such large quantities of cellulose in some reclaims shows the necessity of looking for cellulose in finished rubber goods of a grade in which reclaim might logically be expected. Its presence furnishes a very good indication that reclaim was used in the mixing. The following furnishes a very good illustration of this. Four samples of what appeared to be

high-grade rubber and to be free from reclaim were submitted for test and analysis. Cellulose was determined in them with the following surprising results:

Sample	Cellulose	
	Per cent	Per cent
A.....	1.9	1.88
B.....	2.0	1.97
C.....	1.5	1.5
D.....	2.73	2.74

These results were obviously of great assistance in evaluating the sample, and in addition brought the figure for rubber hydrocarbon in the report nearer to its correct value.

The presence of ground leather in a rubber sample in which cellulose is to be determined does not influence the result obtained upon acetylation. When leather is digested in cresol at 185° C it is detanned. The gelatin that is liberated dissolves in the cresol, while, in the case of vegetable-tanned leather the tannin that is split off is decomposed by the heat into phloroglucinol, carbon dioxide, and metagallic acid.<sup>4</sup> The latter is a dark-colored, non-volatile residue, insoluble in water and acids, and averages 15 per cent of the leather digested. It is easily dissolved when it is washed alternately with a hot 2 per cent solution of sodium hydroxide and a 10 per cent solution of hydrochloric acid. The resulting solution readily reduces potassium permanganate. Since cellulose is not affected by 2 per cent sodium hydroxide solution, it is practical to remove the metagallic acid which was formed from leather present in the mixing by treatment with this solution and then to acetylate in the manner outlined here. When the presence of leather is suspected, it is necessary to wash with a 2 per cent solution of sodium hydroxide and if a deep brown extract is obtained, these washings must be continued, alternating with 10 per cent solution of hydrochloric acid, until the washings are colorless. The pad is then washed with 10 per cent hydrochloric acid, etc., as given in "Details of Method as Proposed."

The determination of cellulose in compound 8 was carried out as given under "Details of Method as Proposed," except that the pad was washed alternately with a hot 2 per cent solution of sodium hydroxide and a 10 per cent solution of hydrochloric acid before acetylating, in order to remove the leather residue, metagallic acid, as explained above.

<sup>4</sup> Allen's Commercial Organic Analysis.



**EXAMPLE 1.—Compound Containing Leather and Substitute**

Compound 8:	Per cent
Smoked sheet.....	45.0
White substitute.....	5.0
Zinc oxide.....	29.0
Leather.....	13.0
Cotton (cellulose, 4.7; moisture, 0.3).....	5.0
Sulphur.....	3.0
	<hr/> 100.0

Cellulose by acetylation, 4.6 per cent.

This test analysis indicates clearly that the presence of considerable amounts of leather does not affect the determination of cellulose.

Other substances which must be considered here are wood, jute, and cork, since these may also be expected in certain types of rubber mixings. When these substances are digested in cresol at 185° C for four hours, they undergo marked changes in composition, and in the case of cork there is decided blackening and change of physical structure. The changes are explained somewhat when one recalls that wood, jute, and cork are considered as incrustated celluloses. They consist of some form of cellulose combined with a noncellulose constituent which may be either of the nature of lignin in the case of the ligno-celluloses such as wood and jute, or a fatty substance in the case of the adipo-celluloses, of which cork is an example. The treatment in cresol at 185° C seems to remove these incrusting substances. In the case of wood and jute there seemed to be complete delignification, for the residue was almost pure white, responded to all the tests for celluloses, and acetylated completely.

The loss in weight that corresponded to delignification ranged from 35 to 55 per cent, and seemed to depend upon the age of the growth from which the fiber was obtained. In the case of cork the loss in weight ranged from 50 to 60 per cent. The residue was very much altered, and no longer exhibited its corky character. This material acetylated incompletely, and left a residue of about 10 per cent of the weight of original sample. Obviously, it is not possible to obtain correct figures for wood, jute, and cork by the method outlined here. The acetylation result represents usually but 50 per cent of the amount of each of these present.

It was found by experiment that the power to delignify wood and jute, and to break down cork structure, did not seem to lie in the cresol itself, but in the temperature at which they were treated, for other liquids were used, and the same phenomenon was noted whenever the temperature rose above 130° C. Below

this temperature there was no noticeable change. Samples of wood, jute, and cork, treated with cresol for 16 hours at 120° C, apparently were unaffected. Wood and jute at the end of this treatment did not give any of the tests for cellulose but responded to those for lignin. When dipped into 1½ per cent alcohol solution of phloroglucinol, and touched with a drop of dilute acid, these fibers were colored red.

Knowing that vulcanized rubber is dissolved in cresol at 120° C in 16 hours, some experiments were carried out in order to ascertain whether a more accurate determination of these fibers could be obtained. It was found that—

TABLE 1

	Wood	Jute	Cork	Leather
	Per cent	Per cent	Per cent	Per cent
Material remaining after 16 hours, immersion in cresol at 120° C.....	98	96	93	90
Material remaining after having been washed with hot 10 per cent solution of hydrochloric acid and water....	95	91	91	70
Acetylation.....	95	90	21	70

The treatment with cresol at 120° C evidently removed some moisture, fats, greases, etc., from these materials, but did not affect the fundamental substance. The washings with a 10 per cent solution of hydrochloric acid and water removed the "water solubles" from the leather. These are usually glucose, Epsom salts, soluble tannin, and run from 5 to 30 per cent of the leather sample. In the cases of wood, jute, and cork it is uncertain just what was removed.

In the cases of wood and jute, it is clear that a comparatively accurate estimation of their quantity can be obtained by using the modified procedure, in which the temperature of 120° C is used in place of 185° C in order to dissolve the rubber.

The acetylation figure includes about 70 per cent of the leather, since all attempts intended to remove it result in an attack upon the lignin of wood and jute, which are sensitive to bromine water, hot sodium hydroxide, etc.

In the absence of wood, jute, and cork an approximation of the amount of leather present can be obtained in the following manner: Digest one sample at 185° C with cresol, remove the leather decomposition products, by means of a 2 per cent solution of sodium hydroxide as given above, and acetylate. The result gives cellulose alone. Another sample is digested at 120° C for 16 hours in cresol, washed with acid, and acetylated. The result

is cellulose and about 70 per cent of the leather. Subtracting from this figure the result obtained above, a result is obtained which can be taken to represent about 70 per cent of the total leather present.

#### EXAMPLE 2.—Approximation of Leather Content

Compound 8:	Per cent
Smoked sheet.....	45.0
White substitute.....	5.0
Zinc oxide.....	29.0
Leather (moisture, 1.2; leather, 11.8).....	13.0
Cotton (moisture, 0.3; cellulose, 4.7).....	5.0
Sulphur.....	3.0
	<hr/> 100.0

Example 2: By acetylation 12.2 per cent. (Calculated from Table 1, this result should be (11.8) (70 per cent) + 4.7 = 12.9 per cent.)

Example 1: By acetylation 12.2 per cent — 4.6 per cent = 7.6 per cent (using 185° C. for digestion in cresol and removing the leather; represents approximately 70 per cent of the leather or 10.9 per cent leather.)

The actual amount of leather is 11.8 per cent, so that 10.9 per cent is to be considered a fair approximation.

Going back to Table 1 it is seen that 21 per cent of cork is acetylated, and that about 70 per cent remains behind on the Gooch pad. This constitutes a partial separation of cork from wood, jute, and leather, since these materials leave no residue after acetylation. The separation, detection, and approximation of cork works out as follows: Acetylation removes wood, jute, and leather, and leaves a residue from the cork which is about 70 per cent of the sample taken. This residue is digested for 1 hour on the steam bath in a 2 per cent solution of sodium hydroxide. It is then filtered through a weighed Gooch, washed alternately with strong bromine water and the hot 2 per cent solution of sodium hydroxide, and finally with a 10 per cent solution of hydrochloric acid. The cork residue is completely removed, and is determined as the loss in weight. It constitutes about 70 per cent of the cork. If no loss in weight is obtained, it means that no cork was present in the sample.

#### EXAMPLE 3

Compound 9:	Per cent
Smoked sheet.....	38.0
Brown substitute.....	5.0
Cork.....	10.0
Zinc oxide.....	42.0
Vaseline.....	2.0
Sulphur.....	3.0
	<hr/> 100.0

By acetylation 1.9 per cent. (Calculated from Table 1, this should be 2.1 per cent.)

The residue after acetylation was treated with a 2 per cent solution of sodium hydroxide and bromine water as given above,



and showed a loss in weight of 6.7 per cent. Considering this as 70 per cent of the cork, the approximation of 9.8 per cent is obtained.

**EXAMPLE 4**

Compound 10:	Per cent
Smoked sheet.....	45.0
Wood pulp.....	7.5
Cork.....	7.5
Pine tar.....	3.5
M. R. X.....	2.0
Zinc oxide.....	20.0
Magnesia usta.....	8.0
Lampblack.....	4.0
Sulphur.....	2.5
	<hr/>
	100.0

By acetylation 9.0 per cent. (Calculated from Table 1, this should have been 8.7 per cent.)

The residue after acetylation when treated to remove the cork, as given above, showed a loss in weight of 5.0 per cent. Considering this as 70.0 per cent of the total, an approximation of 7.1 per cent is obtained.

**EXAMPLE 5.—Stock Containing Jute**

Compound 11:	Per cent
Smoked sheet.....	36.0
Litharge.....	6.5
Zinc oxide.....	43.0
Magnesia usta.....	2.5
Jute.....	10.0
Sulphur.....	2.0
	<hr/>
	100.0

By acetylation 9.0 per cent. (Calculated from Table 1, this should have been 9.0 per cent.)

**EXAMPLE 6.—Stock Containing Wood**

Compound 12:	Per cent
Smoked sheet.....	50.0
Wood pulp.....	13.0
Glue.....	10.0
Zinc oxide.....	20.0
Aluminum flake.....	4.4
Sulphur.....	2.6
	<hr/>
	100.0

By acetylation 12.4 per cent. (Calculated from Table 1, this should have been 12.35 per cent.)

**EXAMPLE 7.—Stock Containing Cotton, Jute, and Leather.**

Compound 13:	Per cent
Smoked sheet.....	40.0
Zinc oxide.....	29.5
Magnesium carbonate.....	4.0
Red oxide of iron.....	4.0
Yellow ochre.....	8.0
Cotton.....	4.0
Jute.....	4.0

## Compound 13—Continued.

Leather.....	4.0
Sulphur.....	2.5

100.0

By acetylation 10.3 per cent. (Calculated from Table 1 this should have been 10.4 per cent.)

In compound 11 and 12, where jute and wood are present alone, a fair determination is possible. In compound 13, where jute, leather, and cotton are all present, a combined figure is obtained which is 1.9 per cent under the total actually present. We have found it impossible to determine each separately. In our opinion the best that can be done is to identify the fibers by satisfactory stains and microscopic examinations, and to determine them collectively, as near as is possible, by acetylation. The rubber content is determined in such cases by the use of rubber solvents at a temperature not over 130° C and the estimation of total fillers.

## V. CONCLUSIONS

1. A method is presented which is readily applicable to the determination of fabric in rubber sheeting, raincoat materials, water-proofed fabrics, spread goods, frictioned and calendered fabrics in general. The results which are obtained by this method have been found to be accurate, by analysis of known compounds. The method has been shown to be useful in the detection and determination of cellulose in reclaims.

2. The presence of leather in mixings is shown not to interfere with determination of cellulose.

3. In the analysis of light, cheap compounds such as rubber soles where wood, jute, cork, and leather may be present, it is found desirable to digest sample at 120° C in cresol for 16 hours, in order to keep these fibers intact. Acetylation obtains 95 per cent of the total wood, 90 per cent of the total jute, 21 per cent of the total cork, and 70 per cent of the leather.

4. The amount of cork present in a mixing can be approximated by removing unacetylated cork residue, and considering this as 70 per cent of the total.

5. In the absence of jute, wood, and cork, it is shown that the amount of leather may be estimated approximately.

6. The problem of separately determining wood, jute, and leather in a mixing has not been solved.

WASHINGTON, October 23, 1919.







